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(Quarterly Report)

INVESTIGATION OF LIGHT SCATTERING IN HIGHLY REFLECTING PIGMENTED COATINGS

National Aeronautics
and Space Administration

IIT RESEARCH INSTITUTE

Report No. IITRI-C6018-14
(Quarterly Report)

INVESTIGATION OF LIGHT SCATTERING IN HIGHLY REFLECTING PIGMENTED COATINGS

February 1 to May 1, 1965

Contract No. NASr-65(07)
IITRI Project C6018

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May 13, 1965

FOREWORD

This is Report No. IITRI-C6018-14 (Quarterly Report) of IITRI Project C6018, Contract No. NASr-65(07), entitled "Investigation of Light Scattering in Highly Reflecting Pigmented Coatings." This report covers the period from February 1 to May 1, 1965. Previous Quarterly Reports were issued on October 11, 1965 (IITRI-C6018-3), January 29, 1964 (IITRI-C6018-6), May 5, 1964 (IITRI-C6018-8), September 5, 1964 (IITRI-C6018-11), December 21, 1964 (IITRI-C6018-12), and March 10, 1965 (IITRI-C6018-13).

Major contributors to the program include Gene A. Zerlaut, Project Leader; Dr. S. Katz and Dr. B. Kaye, theoretical analyses; and V. Raziunas, experimental investigator.

Data are recorded in Logbooks C14085 and C13906.

Respectfully submitted,

IIT RESEARCH INSTITUTE

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ABSTRACT

INVESTIGATION OF LIGHT SCATTERING IN HIGHLY REFLECTING PIGMENTED COATINGS

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The work discussed represents (1) continuing theoretical efforts to elucidate the mechanism of multiple scattering in concentrated pigmented films and (2) continued experimental studies of the optical properties of carefully prepared arrays of silver halide particles. The theoretical work involves both the adaptation of classical light-scattering theory and the generation of random-walk concepts to explain the multiple interaction problem.

Using classical theory, it is shown for a refractive index of 1.44 and an angle of total reflection of 45°, the attenuation cross-section, k, of 1-μ particles for <u>all</u> energy is 0.73 -- emphasizing the preponderance of forward scatter. The attenuation of an incident beam by a multiple array of particles using identical parameters (and assuming no interference by adjacent particles was determined as the number of particles, N, per unit area required to attenuate the incident beam to 1%. In a film of 1-cm^2 area with a mean particle spacing of 10 microns, the film was calculated to be about 2.0 mm thick.

A prerequisite for the development of the random-walk model for a highly pigmented paint is the definition of concepts such as interparticle distance, the significance of an exact knowledge of particle size distribution and the concept of boundary layer conditions. For example, a scientific correlation between pigment particle size distribution and paint properties necessitates an accurate measurement of pigment particle size distribution <u>in the paint system</u>. The significance of an uncertainty relative to particle size when considering the random-walk model is discussed in detail. The discussion of boundary conditions includes (1) surface finish and reflectivity of the boundary between the incident energy and the pigment/vehicle matrix, (2) surface finish and the reflectivity of the boundary between the pigment/vehicle and the body to which the paint film is applied, and (3) the extent and spacial configuration of the paint film.

The reflectance measurements of simulated bimodal coatings indicate that the backscatter intensities due to each particle size are additive and that particles in thin films tend to act as independent scatterers. The spectral back-scatter coefficients for these bimodal suspensions are also presented. Values of back-scatter intensity obtained from the reflectance measurements of the mixtures were consistently less than those predicted from measurements of monodisperse films.

Author

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INVESTIGATION OF LIGHT SCATTERING
IN HIGHLY REFLECTING PIGMENTED COATINGS

I. INTRODUCTION

The principal objective of this program is the application of light-scattering theory to particle arrays in an attempt to explain the scattering behavior of polydisperse pigmented coatings, especially highly reflecting pigmented coatings. In this respect, the program is aimed at a definition of light-scattering parameters associated with the maximum reflection of solar radiation.

Work thus far has involved (1) a review of the applicable light-scattering theory with special emphasis on that portion holding the most promise for application to multiple scattering phenomena, (2) the conception of theoretical approaches and techniques with which to treat the problem of multiple scattering, and (3) the generation of experimental data concerning the optical properties of carefully prepared arrays of silver halide particles dispersed in a matrix.

The studies discussed in Sections II and III are concerned with continuing efforts on (1) the adaptation of classical light-scattering theory to the multiple interaction problem and (2) the adaptation of random-walk techniques to the elucidation of multiple scattering. It has been necessary to define certain concepts as prerequisites for the development of the random-walk model for a highly pigmented, highly reflecting paint film. A general analysis of multiple interaction phenomena and the concept of interparticle distance within a monosized particle cloud were discussed in the last quarterly report (IITRI-C6018-13). The significance of an inexact knowledge of particle-size distribution and the concept of boundary conditions are discussed in this report.

II. APPLICATION OF CLASSICAL THEORY TO MULTIPLE SCATTERING

A. General Discussion

This section describes the continuing study of theoretical film systems through the application of Mie light-scattering theory.

In the last report we examined a number of systems in which a film, described as a collapsed cloud, of an array of monodisperse particles of infinite refractive index embedded in a transparent medium was formed. These particles neither absorb or transmit light; they can only reflect and diffract. Such systems represent a theoretical limit that is approached but never reached in real systems.

We will now give a parallel analysis of particle systems with real refractive index; i.e., all incident light energy is scattered, and none is retained by absorption.

The radial scattering intensity and the attenuation per individual particle are given in Equations 1 and 2.

$$I_\theta = \frac{I_0 \lambda^2 (i_1 + i_2)}{8 \pi^2 R^2} \qquad (1)$$

and

$$I_0 = I_0 e^{-K \pi r^2 N} \qquad (2)$$

In the case of highly reflecting particles, only a small part of the energy incident on the particle undergoes forward scatter, and Equation 2 describes this attenuation very well. However, a large fraction of the energy is scattered in the forward direction by transparent particles. Equation 2 is satisfactory for attenuation of the forward beam in a long path with near-parallel optics, but in the case of a thin film all the energy in the forward direction within the critical angle (the angle of total reflection) is transmitted. This is illustrated in Figure 1 in which the index of refraction* is $1/\sin \Theta$, and all light outside the cone subtended by the angle $2\ \Theta$ undergoes either back scatter or total reflection.

An exact determination of the forward-scatter component is obtained by the integration of the radial scattering function over the solid angle of forward scatter. This is a formidable task, and we will not undertake its rigorous solution. Instead, approximation methods will be used by employing averaged values of the radial scattering terms. Lowan's modified form of Figure 1 is

$$I_{\Theta} = \frac{I_0 \lambda^2 (i_1 + i_2)}{8 \pi^2} \qquad (1a)$$

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* Note that this is the refractive index between the film and air -- not the refractive index of the particle.

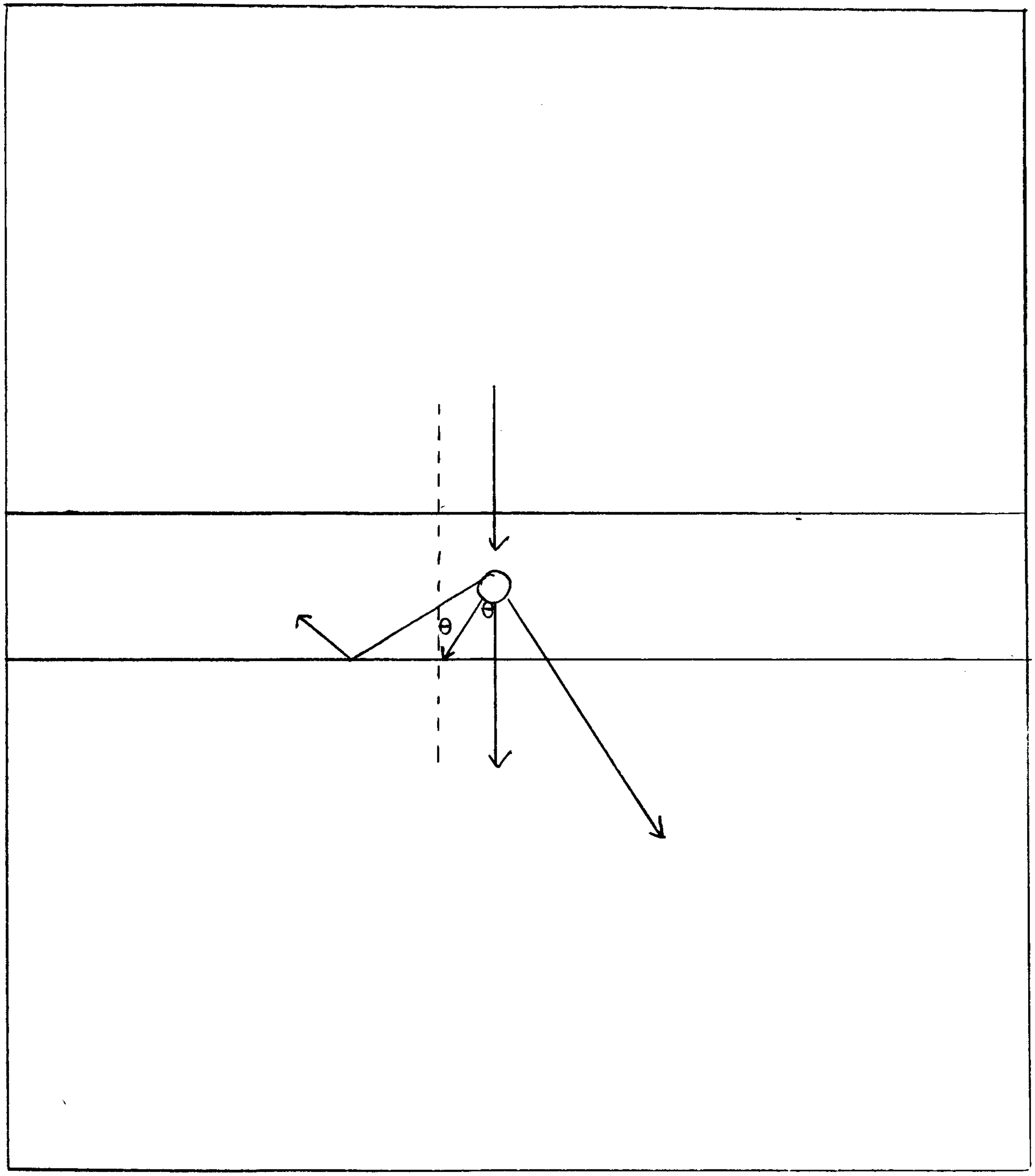


Figure 1

LIMITS OF FORWARD- AND BACKSCATTER IN FILMS

I_0 is the incident energy per unit area; I_θ is the scattered intensity per unit solid angle in the direction θ; and i_1 and i_2 are the angular distribution functions for the two plane-polarized components scattered by a transparent particle illuminated by natural light.

B. Application to a Theoretical Film

Consider first a system of particles embedded in a film. The refractive index of the particles in the medium is 1.44, and the angle of total reflection (Figure 1) is 45°. The forward scattered cone will therefore subtend an angle of 90°, i.e., a solid angle of 1.84 steradians. The following parameters apply.

The scattering cross section, K, has its maximum value of 4.0 when $\alpha = 4.8$ and $m = 1.44$.[1] Consider the case for spheres of 1-μ radius: since $\alpha = 2\ \ r/\lambda$, then $\lambda = 1.31\mu$. By applying a weighted average to the radial scattering data of Lowan[2] the averaged value for i_1 and i_2 in the forward solid angle of 1.84 steradians is about 260. Then from Equation 1a, the scattered intensity per unit solid angle in the forward cone is 5.6×10^{-8}, where the incident energy is unity per unit area, or 10.3×10^{-7} for a solid angle of 1.84.

[1] LaMer ans Sinclair, Chem. Rev., 44, 245 (1949).

[2] Lowan "Tables of Scattering Functions for Spherical Particles," National Bureau of Standards, 1948.

Also, the scattering cross section of a particle of 1-μ radius is $K \pi r^2 = 12.6 \times 10^{-8}$ cm^2, and the total scattered intensity is therefore 12.6×10^{-8} energy units. Then the backscattered energy by one particle of 3.14×10^{-8} cm^2 cross section will be $(12.6 - 10.3) \times 10^{-8} = 2.3 \times 10^{-8}$.

Thus the attenuation cross section, K, of a particle for <u>all</u> energy, which is not the same as the cross section for the incident beam alone, is given by $2.3 \times 10^{-8} / 3.4 \times 10^{-8}$ and, in this case, is 0.73. This low figure emphasizes the preponderance of forward scatter with this material.

Now the attenuation of an incident beam by a multiple array of particles can be computed. The parameters are the same as those used above, and no interference by adjacent particles is assumed. Then the attenuation as stated in Equation 2 is

$$2.303 \log I_0/I = K^1 \pi r^2 N.$$

By replacing K^1 and r by their indicated values (0.73 and 1, respectively), we can determine the number of particles, N, per unit area required to attenuate the incident beam to 1%:

$$2.303 \log 100 = 0.73 (10^{-4}) \pi^2 N$$

Solving,

$$N^1 = 2.0 \times 10^8 \text{ particles per cm}^2.$$

In a film of 1 cm^2 area with a mean particle spacing of 10μ, i.e., 5 particle diameters spacing between adjacent particles, the film will be about 2.0 mm thick.*

In the next report, we will extend these calculations to indicate the variation of the scatter as the other parameters -- wavelength, particle size, and refractive index are varied. Also we will examine the scattering for some types of nonspherical systems.

---

*This calculation was made with no attempt to correct for the boundary condition in which particles are very close to the totally reflecting surface.

III. RANDOM-WALK TECHNIQUE FOR STUDYING MULTIPLE SCATTERING

A. Development of Random Walk within a Collapsed Cloud

In developing the random-walk model for the condensed cloud, i.e., for the paint film, it is necessary to know exactly what is meant by diffuse light, particle size, refractive index, reflection, and nature of the boundaries of the paint film. When the paint literature is examined closely it is found that many of their terms are used loosely. It is necessary to define strictly the concepts used. A prerequiste for the development of the random-walk model is a critical discussion of these concepts. Furtheremore the testing of any predicted theory requires knowledge of the exact nature of the experiments that can be performed and the relationship between ideal and actual measurements.

A discussion of boundary concepts and the meaning of particle size follows. The exact specification of particle size is also necessary in the extraction of information from published data on light-scattering systems and in the simulation of random screens for the Type II random walk (i.e., the collapsed cloud).

B. Specification of Pigment Size

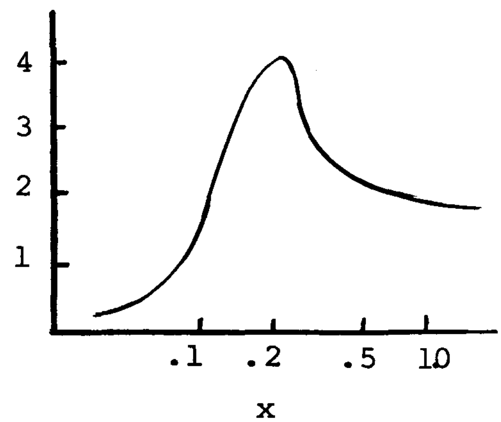
In an excellent review article on the relationship between the particle size of pigments and the properties of the paint made from them, Newman[3] made these comments. "If one wishes to make a scientific correlation between pigment particle size and paint properties, it is necessary to be able to measure with some reasonable degree of accuracy quantities involved in the proposed correlation. Unfortunately, the scientific acceptable knowledge both of the pigment particle sizes and of really significant paint properties still leaves much to be desired."

Although these remarks were made almost 20 years ago, they seem to be very relevant to the current state of knowledge and practice in the paint industries. In particular, it is not sufficiently realized that the precision with which a particle size can be defined may be the limiting factor, when applying light-scattering theory to the study of the optical behavior of paints. For example, consider the curve shown in Figure 2. In the literature this type of curve has been suggested as an effective universal curve to be used when considering pigment optics.[4] The merits of this curve

---

[3] Newman, A.C.C., "Particle Size in Relation to the Use of Pigments in Paints," Supplement to Trans. Inst. of Chem. Eng., 25, 88 (1947).

[4] Barnett, C.E., Ind. Eng. Chem., 41, 272 (1949).



K = Extinction Coefficient

x = $d \frac{m^2-1}{m^2+2}$, where m is relative refractive index

Figure 2

CLAIMED UNIVERSAL EFFECTIVE EXTINCTION CURVE
FOR PIGMENT PARTICLE

as a valid measure of optical scattering power will be discussed later. However, it should be noted that its interpretation should be treated with great caution. But, leaving the question of its validity open at this stage of the discussion, let us consider the difficulties of applying it even if it were established as the operative, effective function.

Consider the problem of predicting the performance of a pigment by using this curve. In the July 16, 1963 issue of Chemical Processing it was claimed that hydrated alumina could be used as a pigment or filler in paint system. The measured characteristics of the powder were quoted as follows:

Average Particle Size, μ	Method
0.12	B.E.T. Method
0.39	Fisher subsieve sizes
0.60	M.S.A. centrifuge
0.30	Electron microscope

In applying the light-scattering curve of Figure 2 (even making the daring assumption that the average particle size can be used to calculate the overall properties of the paint), which value of the diameter should be used? The two extreme estimates differ by a factor of 5, and the four estimates would locate at very different regions on the scattering curve.

The other aspect of this problem is the difficulty of correlating reported data on the light-scattering properties of small particles. It is common practice to report data by drawing graphs of some measured quantity such as extinction coefficient versus a particle diameter. However, the significance of the measured diameter quoted is not always apparent. For example, consider the curves in Figure 3. These curves represent data from light-transmission data reported by Prof. Andreasen and co-workers[5] from their studies of barium sulphate suspensions. The variable $\bar{\mathcal{E}}$, is defined as the specific extinction and is related to the extinction coefficient. The significance of this transmission data will be discussed when reviewing transmission measurements reported for suspensions. The important point to notice is that only a careful reading of the text of the Andreasen paper reveals that the particle-size parameter used in plotting the data is the length of the cube volume equal to that of the sphere of equal Stokes' diameter. Let this length be k.

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[5] Andreasen, A.H.M., K. Krebs, N.Aa, Dalsgaard Pederson, E. Damradt Petersen and B. Kjaer, Suppl. Trans. Chem. Eng., 25, 4 (1947).

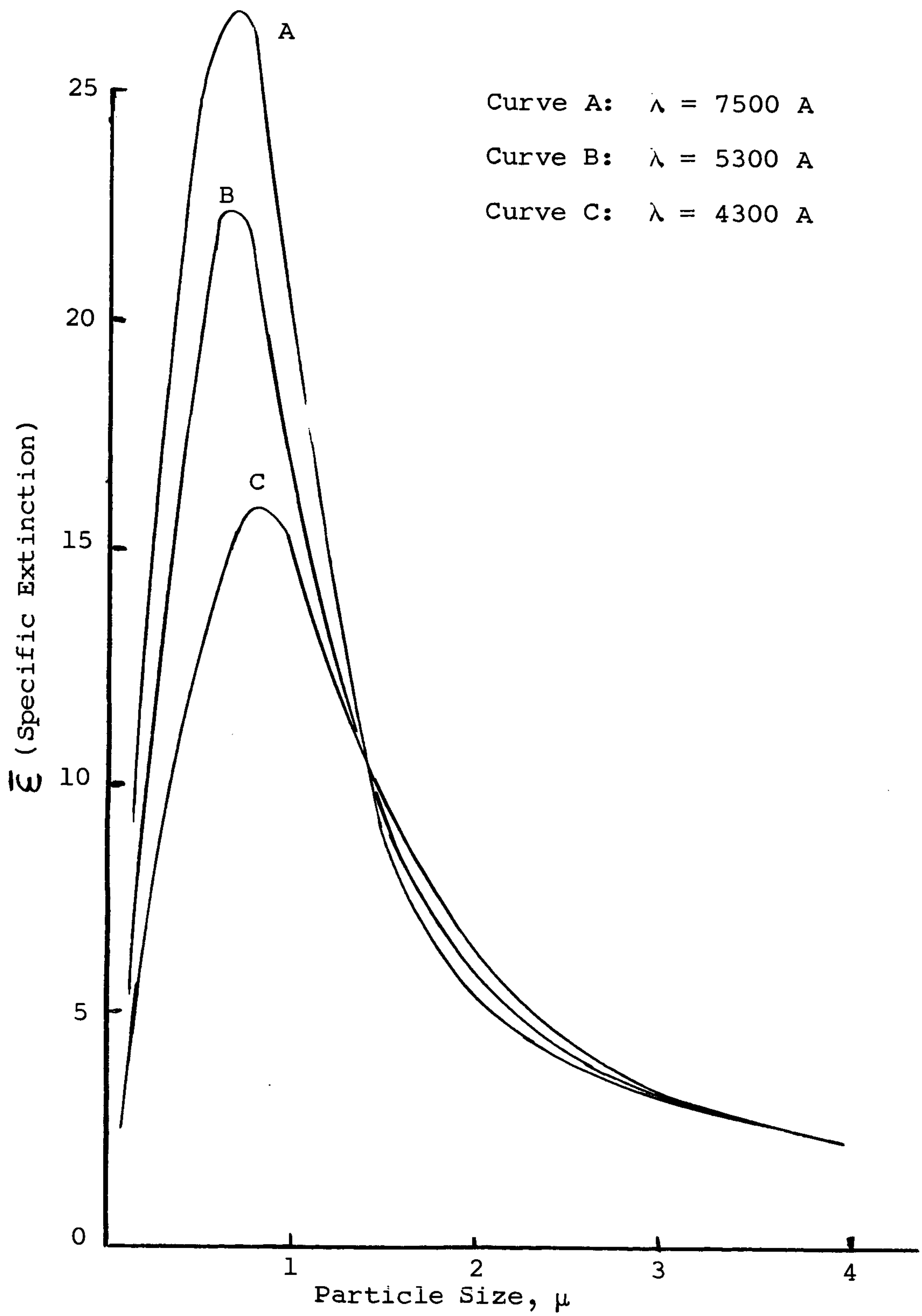


Figure 3

LIGHT-SCATTERING PROPERTIES OF BARIUM SULPHATE PRECIPITATES

By definition,

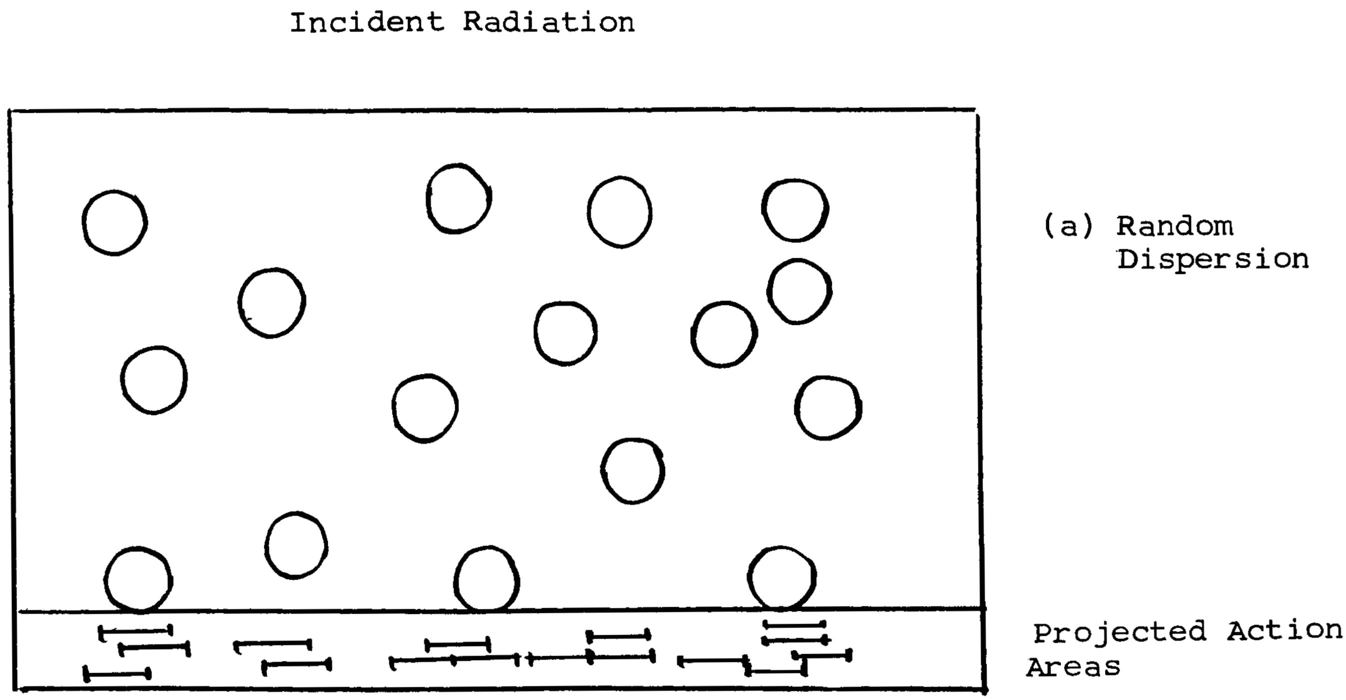
$$K^3 = \frac{1}{6} \pi d^3$$

$$\frac{k}{d} = \sqrt[3]{\frac{\pi}{6}} = \sqrt[3]{0.525} = 0.807$$

Therefore, without a full realization of the exact definition of particle size used, a superficial location of the peak of the transmission could have been 20% off, if the Stokes' diameter had been used as the parameter.

In considering the random screens exposed on sectioning a paint film, the question of pigment size specification becomes accute. If the reasons for the discrepancies between the various pigment size estimates quoted for the alumina could be eliminated, what could be said about the pigment when it is in the paint film? In the study of sections through paint films analytical techniques for determining the state of dispersion and the spatial configuration of the pigment locations are almost nonexistent.

To understand the importance of this problem for the random-walk model, consider the systems illustrated in Figure 4, which illustrates the problem in two dimensions of considering the effect of distribution of a given pigment.



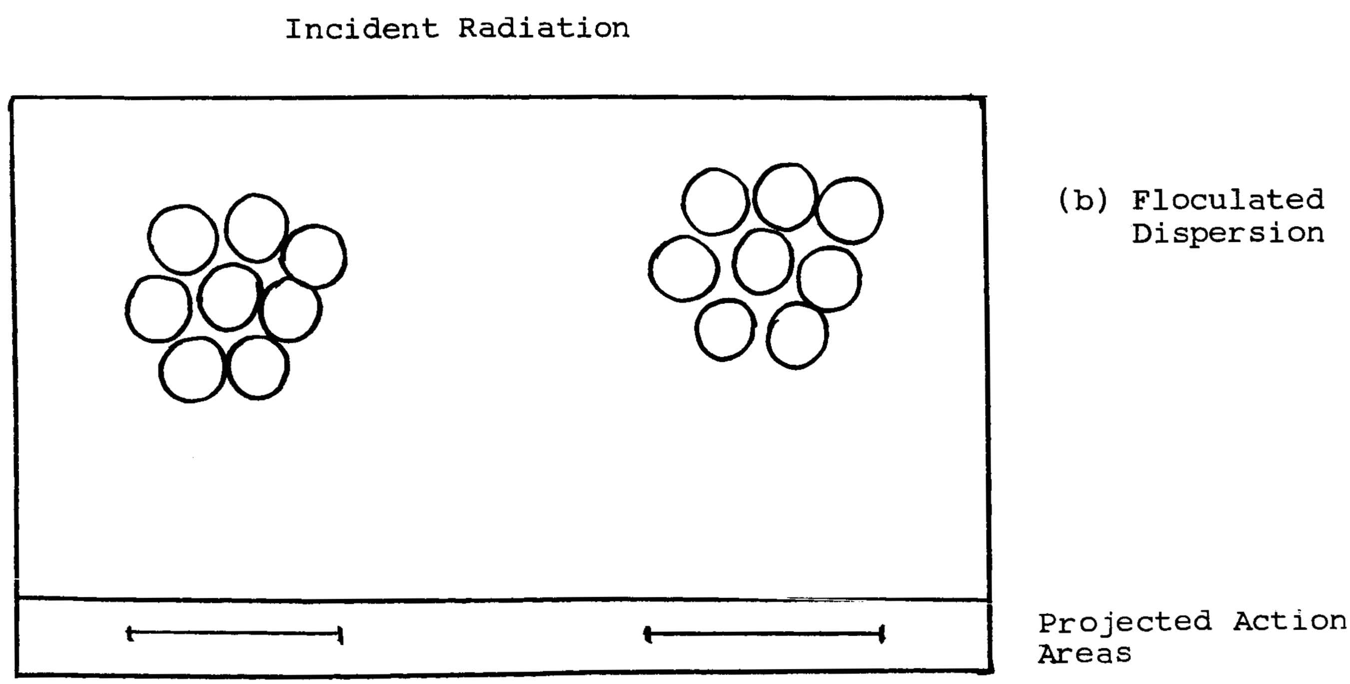


Figure 4

DIFFERENT DISPERSION STATES WITHIN A PIGMENT FILM

In Figure 4a a number of circles are set at random. In Figure 4b these are collected into random clusters.

The grouping of the particles has a twofold effect. First, it reduces the area of the film in which pigment particles are available for scattering interaction. Even if a very crude estimate of interaction area is made by projecting interaction areas along the base line, it can be seen that the clusters are less effective. It is not sufficient to regard the clusters as forming particles of larger diameter. The cluster has a higher effective absorption coefficient, because the internal porosity of the cluster tends to oscillate back and forth, and the low absorption coefficient of the single particle becomes important because of the many possible paths within the cluster.

It has sometimes been noted that a filler improves the opacity of an active white pigment. This improvement could be due to the fact that random mixing of the larger inert filler particles prevents large clusters of pigment from forming by filling in the interstitial spaces available. One way to improve the performance of paint may be a mechanical process for ensuring random efficient dispersion within the film. In testing the performance of any film from any theory, however, the exact nature of the distribution within a paint film must be known. In the next report a few suggestions will be given on possible techniques of determining particle properties within the film.

C. Boundary Conditions in the Boundary of Refectivity of Paint Films

An ultimate aim of the program is the explanation of the reflectivity of paint films. To enable any postulated optical theory of paint behavior to be applied to any paint-film system, the boundary conditions of the paint film must be known. The important boundary conditions are:

(a) Surface finish and reflectivity of the boundary between the incident energy and the pigment/vehicle matrix

(b) Surface finish and reflectivity of the boundary between the pigment/vehicle and the body to which the paint film is applied

(c) The extent and spatial configuration of the paint film.

The surface finish of the boundary surfaces are important because they effect the energy entry, energy escape, and directional properties of the radiation within the paint film. The paint industry is concerned with the surface finish of a paint, but only gross qualitative properties such as the gloss or matt nature of a surface are measured.

In the study of the interaction of waves with a boundary, it is generally recognized that the surface is smooth if irregularities are small compared with the wavelength of light. However, there is little information on how surface irregularities affect radiation incident on a surface or on how large a smooth area must be before regular reflection occurs.

In a textbook on optics, J. Strong[5] quotes data for infrared reflection from brass plates of various roughness. Figure 5 shows that the energy entering the brass plate depended greatly on the surface finish. On a superficial level it can be argued that, since a smooth surface rejects a higher amount of energy than a rough one, the aim should be to have a smooth surface finish. In this manner the pigment/vehicle reflecting matrix will have less energy to cope with.

However, a little consideration shows that this argument is too simple. All the energy returned to the surface by the pigment particles has to pass through this surface to be expelled from the system. The very fact that the smooth surface is an efficient barrier to incident energy means that scattered energy returning from the pigment particles encounters an efficient radiation barrier. It is conceivable that a rough surface may be more effective when averaged over many radiation-transfer events.

A second property of a rough surface that could increase the overall reflectivity of the surface is that directional

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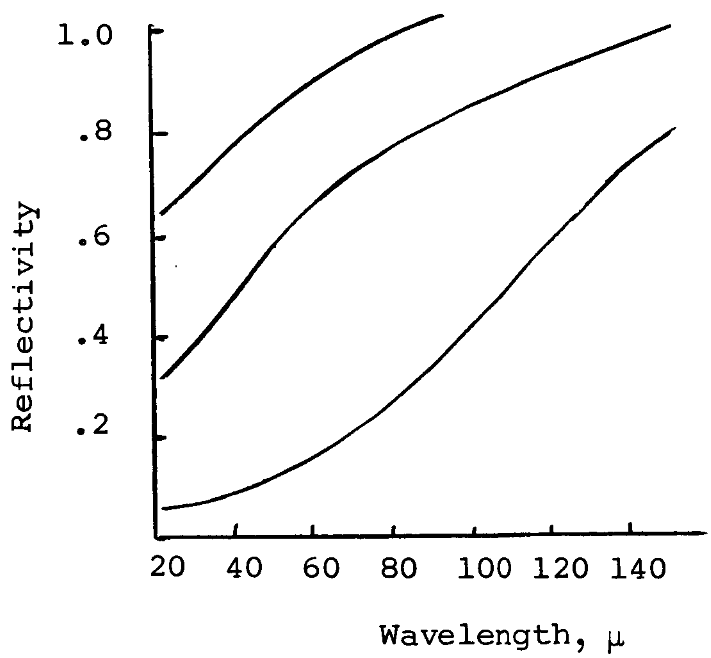
[5] Strong, J., Concepts of Classical Optics, Freeman & Co., London, 1958, p. 279.



A - Brass plate polished with fine abrasive powder

B - Brass plate polished with medium-grade abrasive powder

C - Brass plate polished with coarse abrasive powder

Figure 5

REFLECTIVITIES OF BRASS PLATES
OF DIFFERENT SURFACE QUALITY

properties of the incident radiation could be changed; i.e., part of the incident radiation would become diffuse and would be spread over a greater area of the pigment film, which therefore would be utilized more efficiently. Without quantitative information on the reflectance of directed and diffuse light from rough surfaces and also on the reflective power of smooth surfaces for incident diffuse radiation, it is not possible to develop quantitatively the collapsed cloud, random-walk model. It is suggested that relatively simple experiments could provide information that could be used to calculate the efficiency of paint films as radiation reflectors. The suggested set of experiments is illustrated in Figure 6. Directional reflection measurements are made with a smooth film, and then the experiments are repeated after the film has been roughened by rubbing with an abrasive powder. The diffuse light experiments could be carried out by using an integrating sphere.

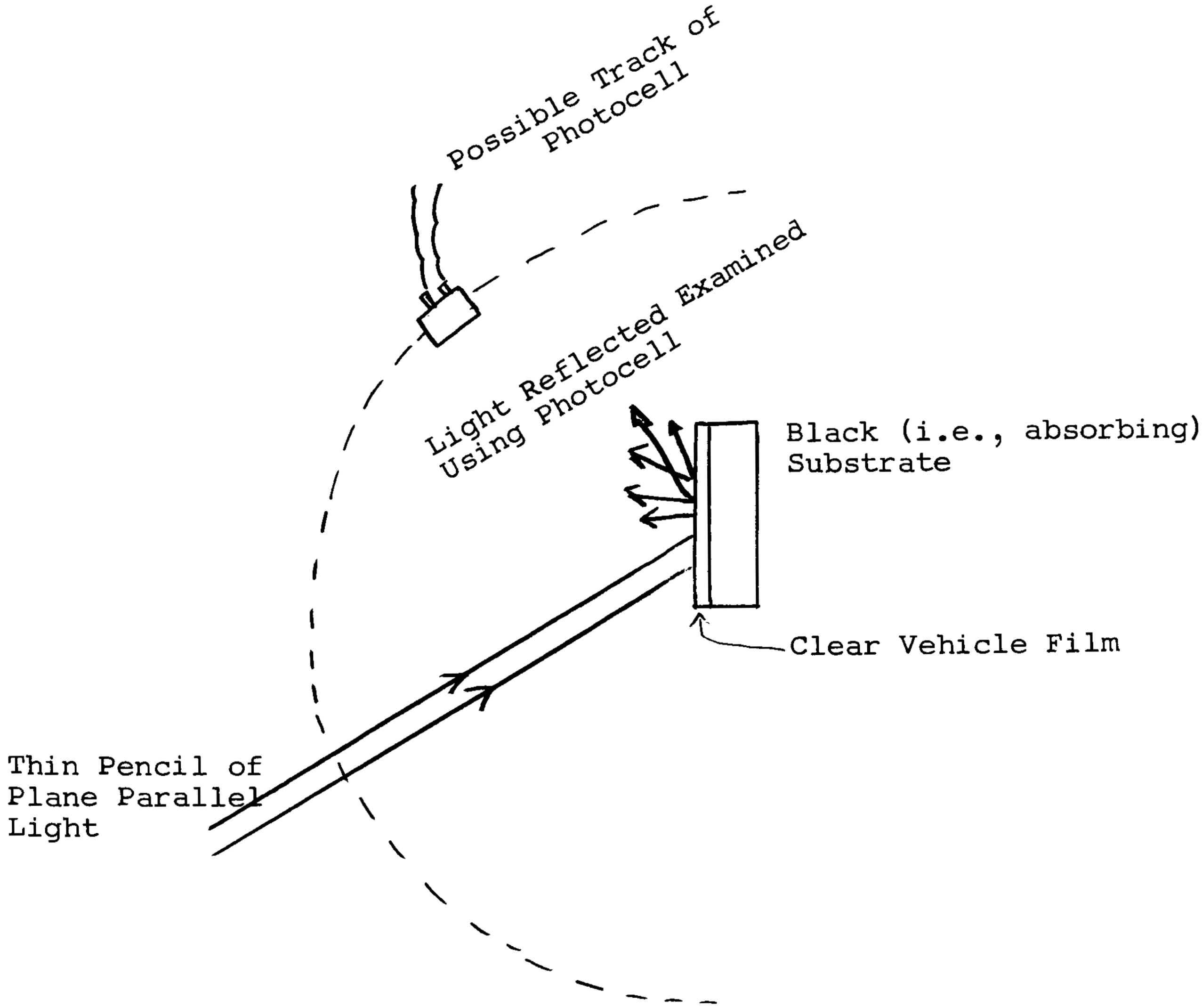


Figure 6

SUGGESTED TECHNIQUE FOR MEASURING REFLECTANCE OF ROUGH SURFACES

IV. EXPERIMENTAL STUDIES

A. Introduction

Studies of transmittance and reflectance properties of concentrated thin films of silver bromide suspensions in gelatin were continued. During a past report period (Report No. IITRI-C6018-12) several sets of thicker semitransparent films were prepared for direct comparison, so that both the transmittance and reflectance could be measured on the same films. Sets of three films were compared in each case. Two films obtained from monodisperse suspensions were compared with the film of the bimodal mixture. A detailed description of the methods of comparison and the experimental data on the films is given in previous reports. The reflectance measurements were made against a magnesium carbonate block.

B. Reflectance of Simulated Bimodal Coatings

During this report period attempts were made to relate transmittance and reflectance of the same films directly. The transmitted light represents the fraction of the light beam that has not been deflected due to scattering (neglecting molecular absorption). Thus, the optical density (O.D. = ln 1/T) is a direct measure of the total scattering, which is the light scattered in all directions. A hemispherical reflectance measurement represents the integrated intensity of the light backscattered toward the incident beam; therefore the reflectance measurement can be related to optical density in extinction (optical density) units as follows:

$$\text{Backscatter intensity} = \ln \frac{1}{100\% - \%\ \text{reflectance}}$$

The ratio of backscatter intensity to the optical density represents the fraction of total scattered energy that is reemitted in the direction of the incident beam. Thus, the optical density and backscatter intensity can be related as follows

(a) Optical density obtained from parallel transmittance measurements:

$$\text{O.D.} = \ln \frac{1}{T} = \frac{1}{100\% - \%\ \text{light scattered in all directions}}$$

(b) Backscatter intensity obtained from the reflectance measurements

$$\text{B.I.} = \ln \frac{1}{100\% - \%\ \text{backscattered light (reflectance)}}$$

(c) Backscattering coefficient $= \dfrac{\text{B.I.} \times 100}{\text{O.D.}}$

Two modes of illumination were used in the reflectance measurements. In one case a parallel beam of monochromatic light at normal incidence was used for illumination, and the integrated hemispherical reflectance was measured. In the other case diffuse undispersed light was used for illumination, and the normal component of the reflectance spectrum was measured.

The reflectance measurements were identical for both modes of illumination; this would be expected for backscatter due to isotropic particles.

The reflectance spectra are given in Figures 7-9. The transmittance spectra for the same films were given in Report No. IITRI-C6018-12. The corrections for varying film thickness were also given.

In calculating the values of backscatter coefficient (Figures 10-12), the backscatter intensity values due to the gelating blank were subtracted from the backscatter intensity values of silver bromide suspensions in gelatin in order to compensate for the Fressnel reflections due to the gelatin. The backscatter coefficients given in Figures 10-12 were calculated as follows:

$$\text{Coefficent backscatter} = \frac{\text{B.I. (Gelatin + AgBr) - B.E. (Gelatin) x 100}}{\text{O.D.}}$$

No compensation for Fressnel reflections was necessary in the optical density values, because the transmittance measurements were made with a gelatin blank in the reference beam.

The reflectance measurements indicate that the backscatter intensities due to each particle size are additive and therefore in backscattering, the particles in thin films tend to act as independent scatterers. Values of backscatter intensity obtained from the measurements of the mixtures were consistently less

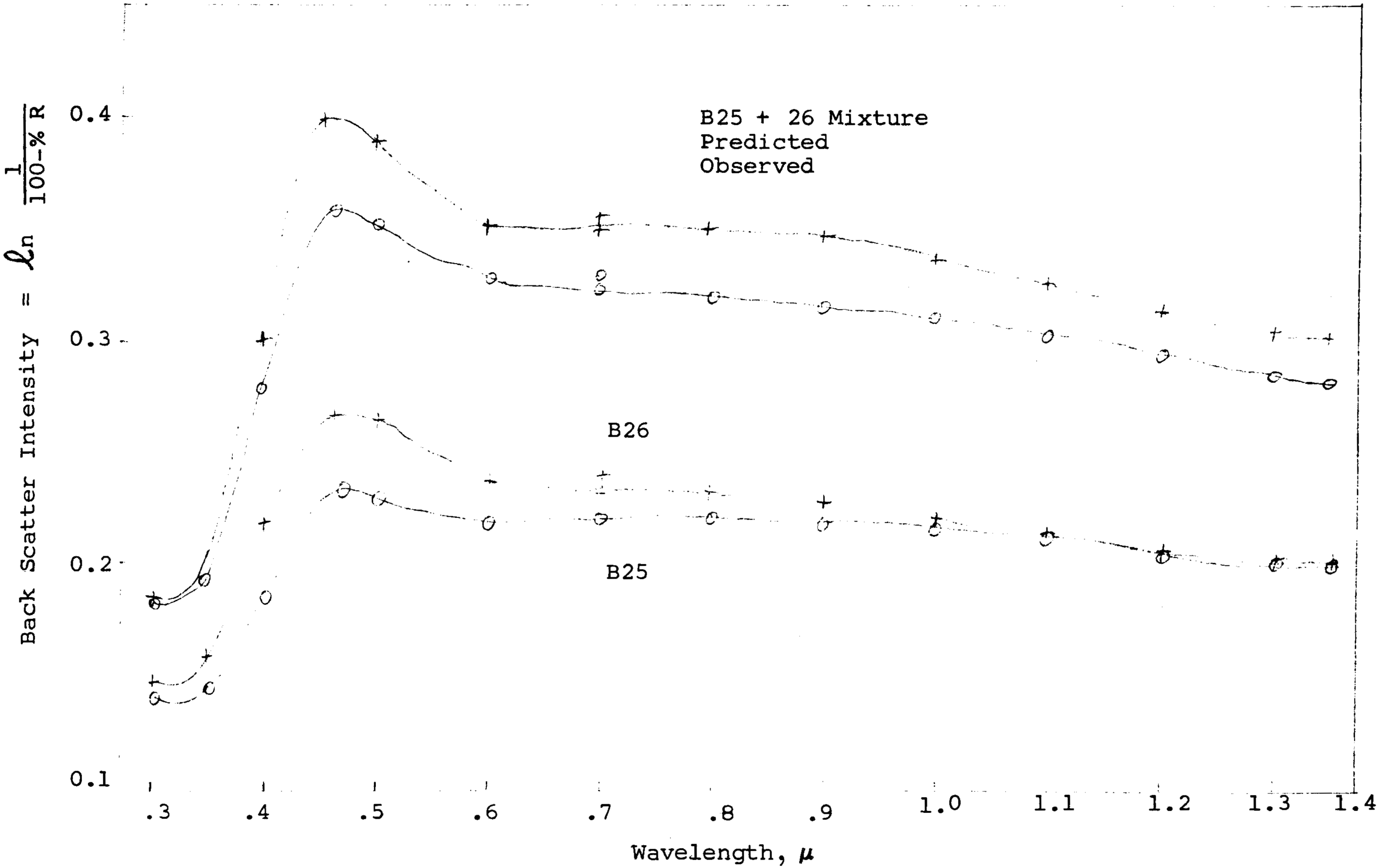


Figure 7

THE BACKSCATTER INTENSITY OF BIMODAL SUSPENSION

(Batches 26 and 25)

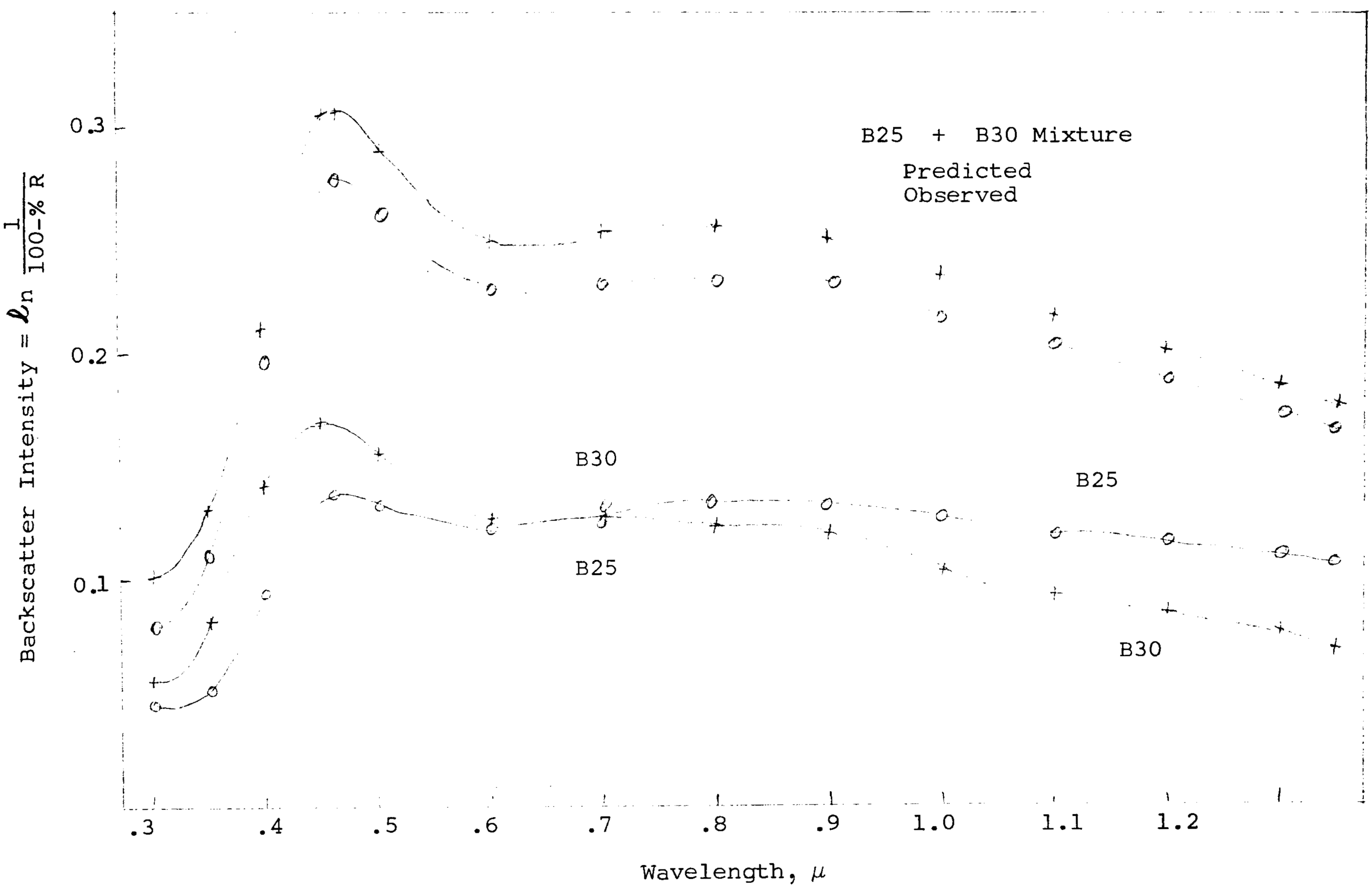


Figure 8

THE BACKSCATTER INTENSITY OF BIMODAL SUSPENSION
(Batches 25 and 30)

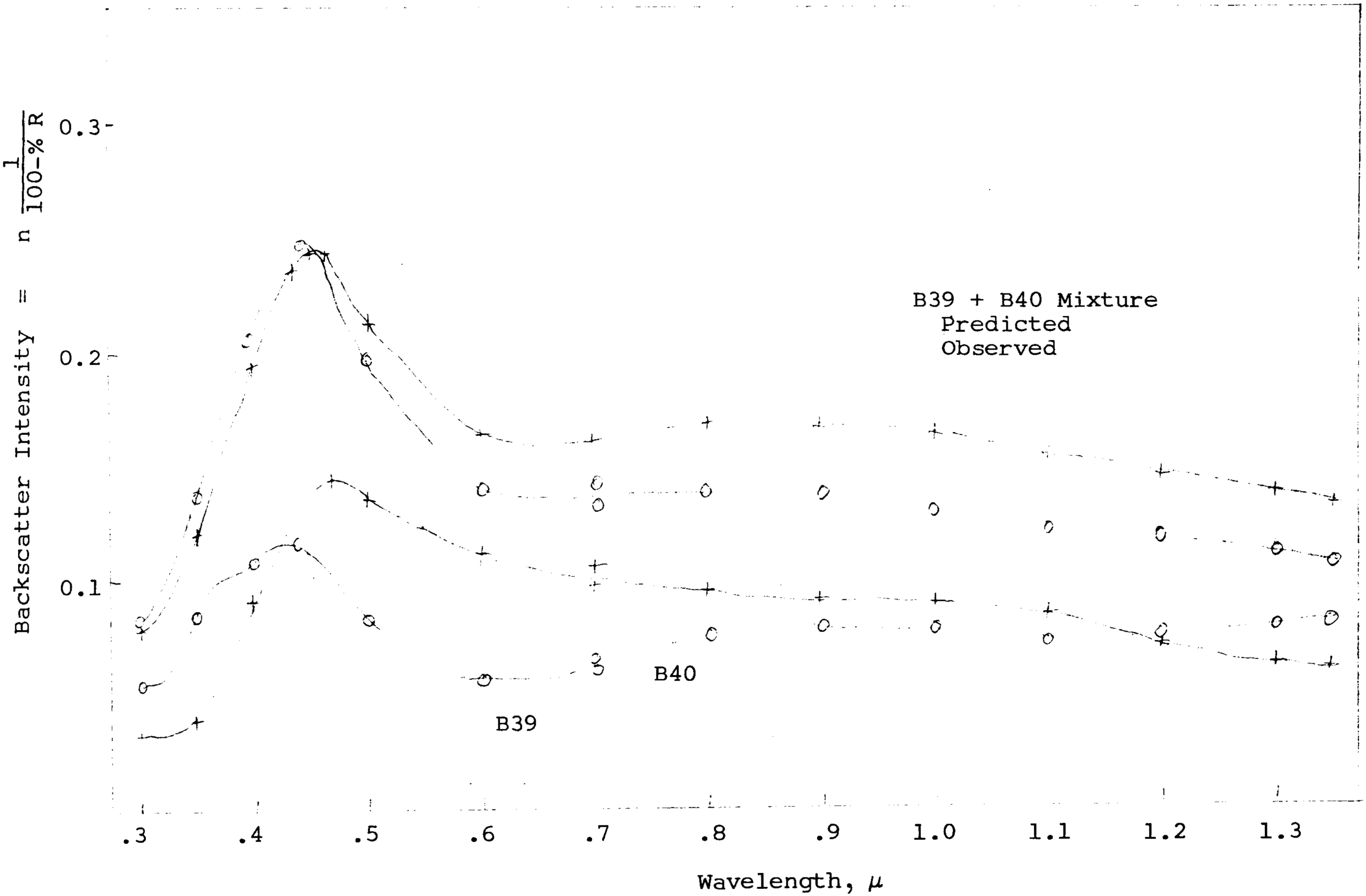


Figure 9

THE BACKSCATTER INTENSITY OF BIMODAL SUSPENSIONS
(Batches 39 and 40)

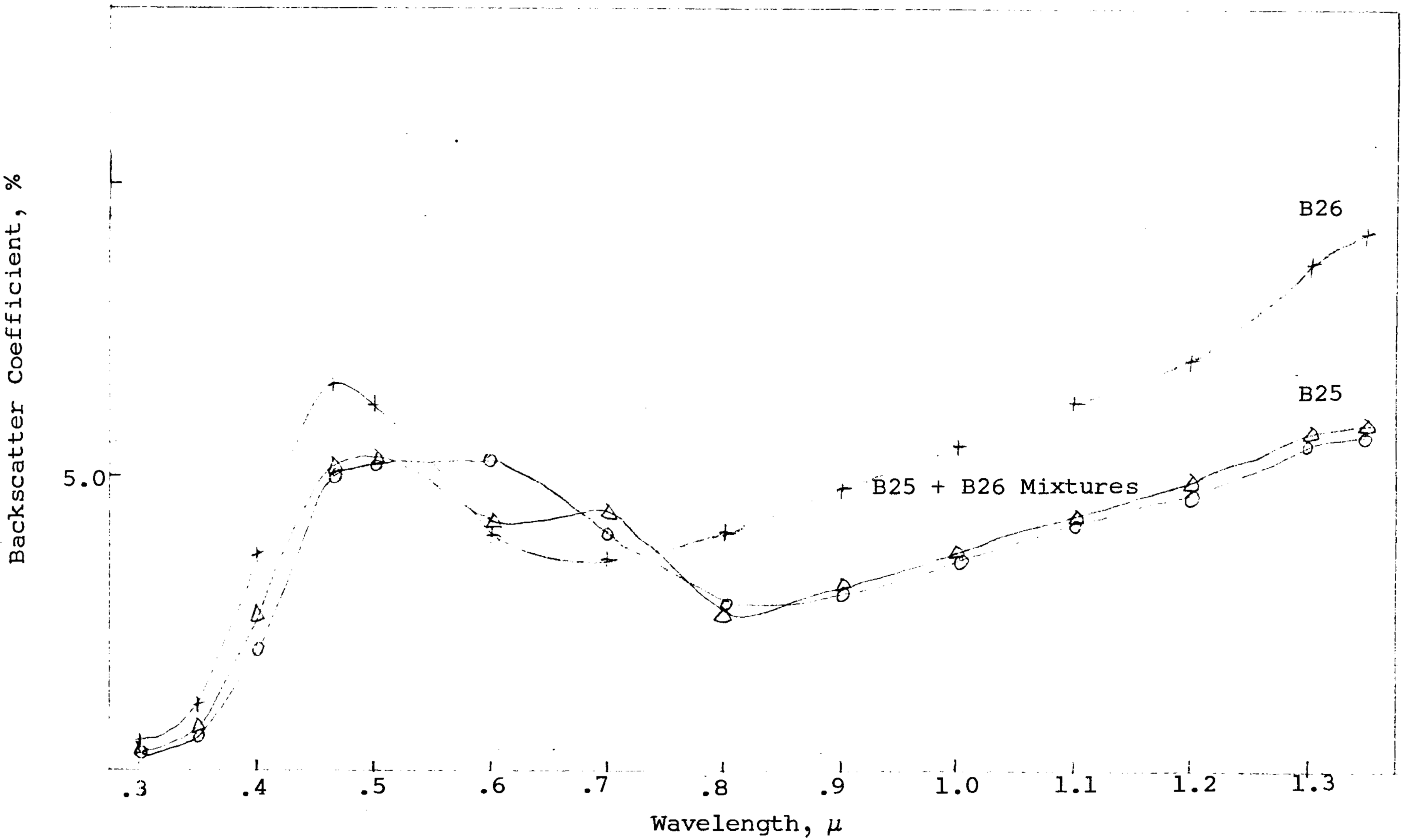


Figure 10

THE BACKSCATTER COEFFICIENT OF REFLECTING FILMS

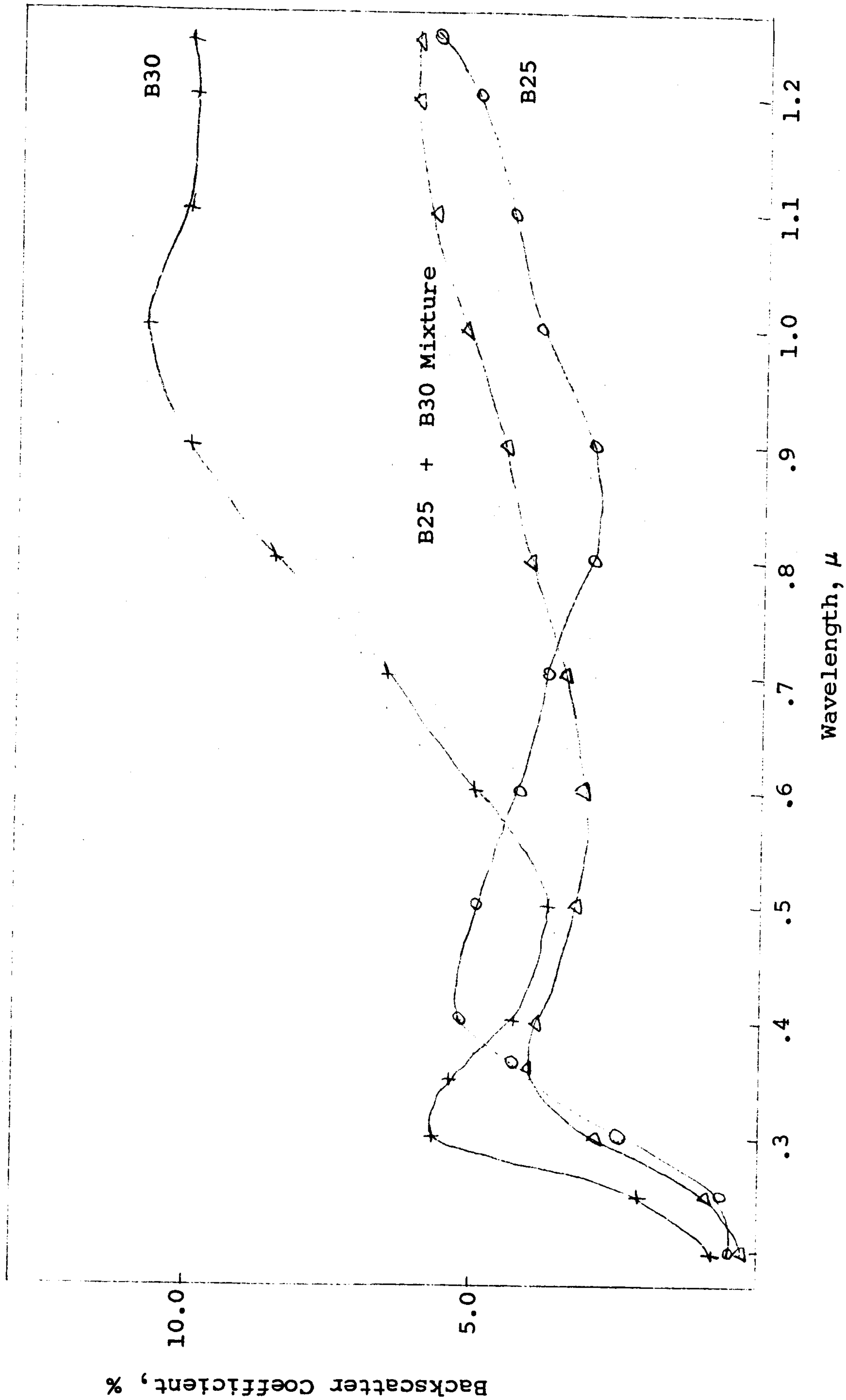


Figure 11

BACKSCATTER COEFFICIENT OF REFLECTING FILMS

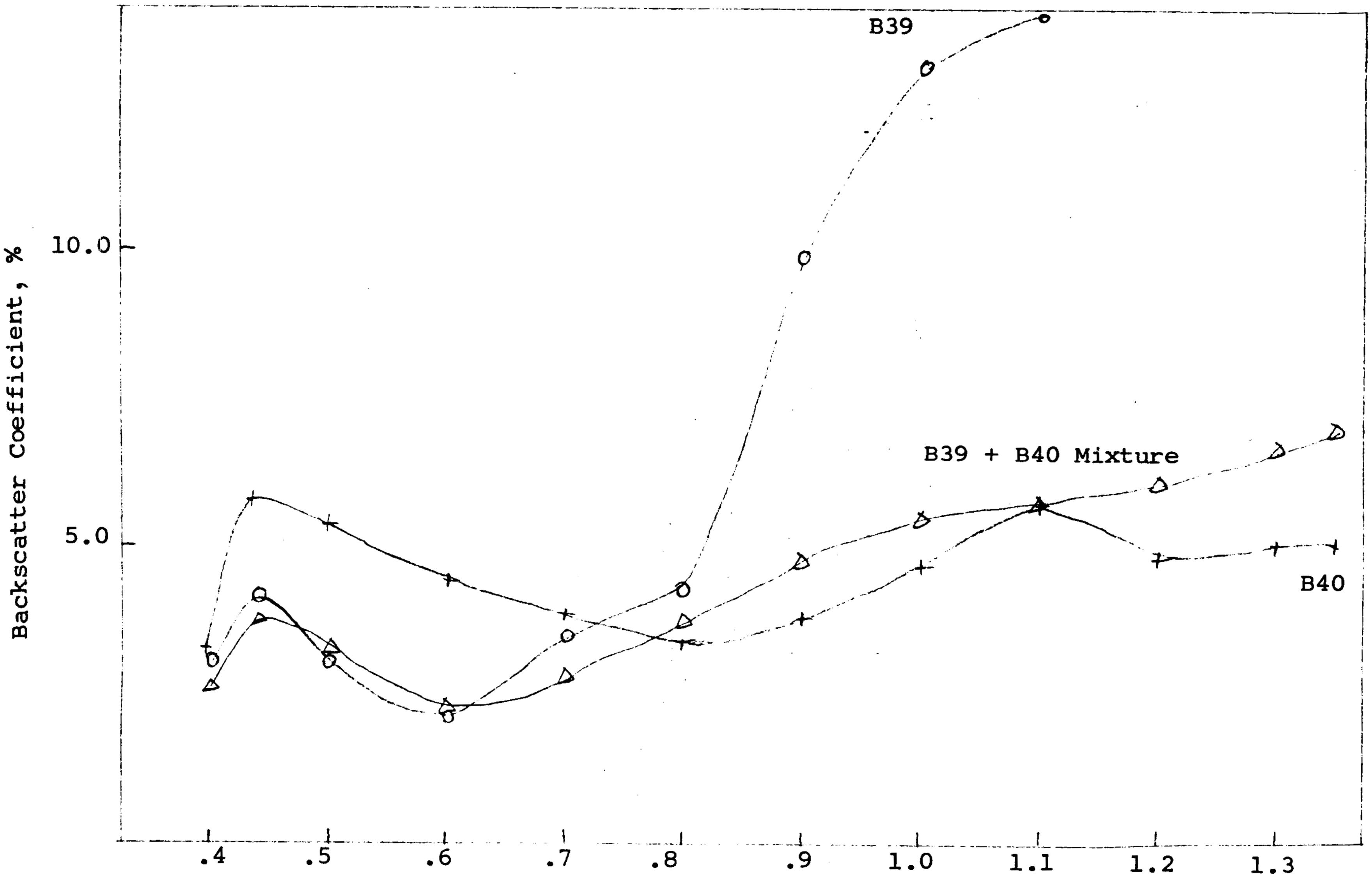


Figure 12

THE BACKSCATTER COEFFICIENT OF REFLECTING FILMS than those predicted from measurements of monodisperse films. The above observation could be due to experimental errors, or it may indirectly support the validity of our previously proposed ideal coating that consists of thin layers (Report No. IITRI-C6018-11).

C. Future Work

Attempts are being made to develop experimental procedures for obtaining thicker coatings of high uniformity. Such coatings of consistently variable thickness would have higher reflectivities and would give more accurate reflectance measurements. The depth of energy penetration could be determined by varying the thickness. The present experiments with monodisperse and bimodal coatings will be continued to check the validity of our observations.

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